

Fig.1A

Step 1

Preparation of silver nitrate solution (solution A)

A solution of 2.1 g silver nitrate in 1 ml water is adjusted to 500 ml with isopropanol.

Step 2

Preparation of sodium acetate solution (solution B)

A solution of 0.4 g sodium acetate in 0.5 ml water is adjusted to 500 ml with isopropanol.

Step 3

Preparation of liquid catalyst

Solution A is mixed with solution B at ordinary temperature, and a white suspended matter (silver acetate) is formed.

Step 4

Catalyst purification operation (No. 1)

The white suspended matter is filtered off with a millipore filter (0.45 μ m) and is removed.

Step 5

Desulfurization with X-rays

By irradiation with X-rays (X-rays containing the absorption edge wavelength (5.018 \AA) of sulfur), sulfur contained as the impurity is changed into a silver compound containing silver sulfide. The time of irradiation with X-rays is 1 hour (for 15 ml container). The sample is then left.

Fig.1B

Step 6

Catalyst purification operation (No. 2)

The black precipitates are filtered off through a millipore filter (0.45 μ m), and sulfur contained as the impurity is removed as silver compound (mainly containing silver sulfide).

Step 7

Dissolved oxygen-removing operation

The liquid catalyst is bubbled with N_2 for 20 minutes, to remove dissolved oxygen.

Step 8

Liquid catalyst stabilization operation

10 ml benzaldehyde is added to the liquid catalyst, to reduce oxidation of the liquid catalyst.

Step 9

Storage of the liquid catalyst

The liquid catalyst is stored in a cool and dark place.

Fig.2

Step 1

Dissolved oxygen-removing operation

The liquid catalyst is bubbled with N₂ for 20 minutes, to remove dissolved oxygen.

Step 2

Sample collection

5 ml sample is collected in the liquid sample container.

Step 3

Liquid catalyst collection

2 ml liquid catalyst is placed in the sample-containing container and stirred sufficiently.

Step 4

Preliminary reaction of the sample

By irradiation with X-rays (X-rays containing the absorption edge wavelength (5.018 Å) of sulfur), a very small amount of sulfur in the sample is converted into a silver compound (mainly containing silver sulfide). Silver-reacting elements other than sulfur are also converted into silver compounds. These are co-precipitated with silver in the bottom of the measurement container. The time of irradiation with X-rays shall be 30 minutes/sample.

Step 5

Extraction of the object component (sulfur compound)

In the presence of co-sedimented silver compounds and silver, scattered X-rays are increased and the absorption of a characteristic X-ray of S occurs, thus making analysis of a very small amount of sulfur difficult. Accordingly, ammonia or aldehyde is added to the sample and then left at a constant temperature (30 °C) for about 20 hours. By this operation, silver compounds and silver other than the object component are dissolved, diffused and removed from the sample measurement surface.

3
Fig

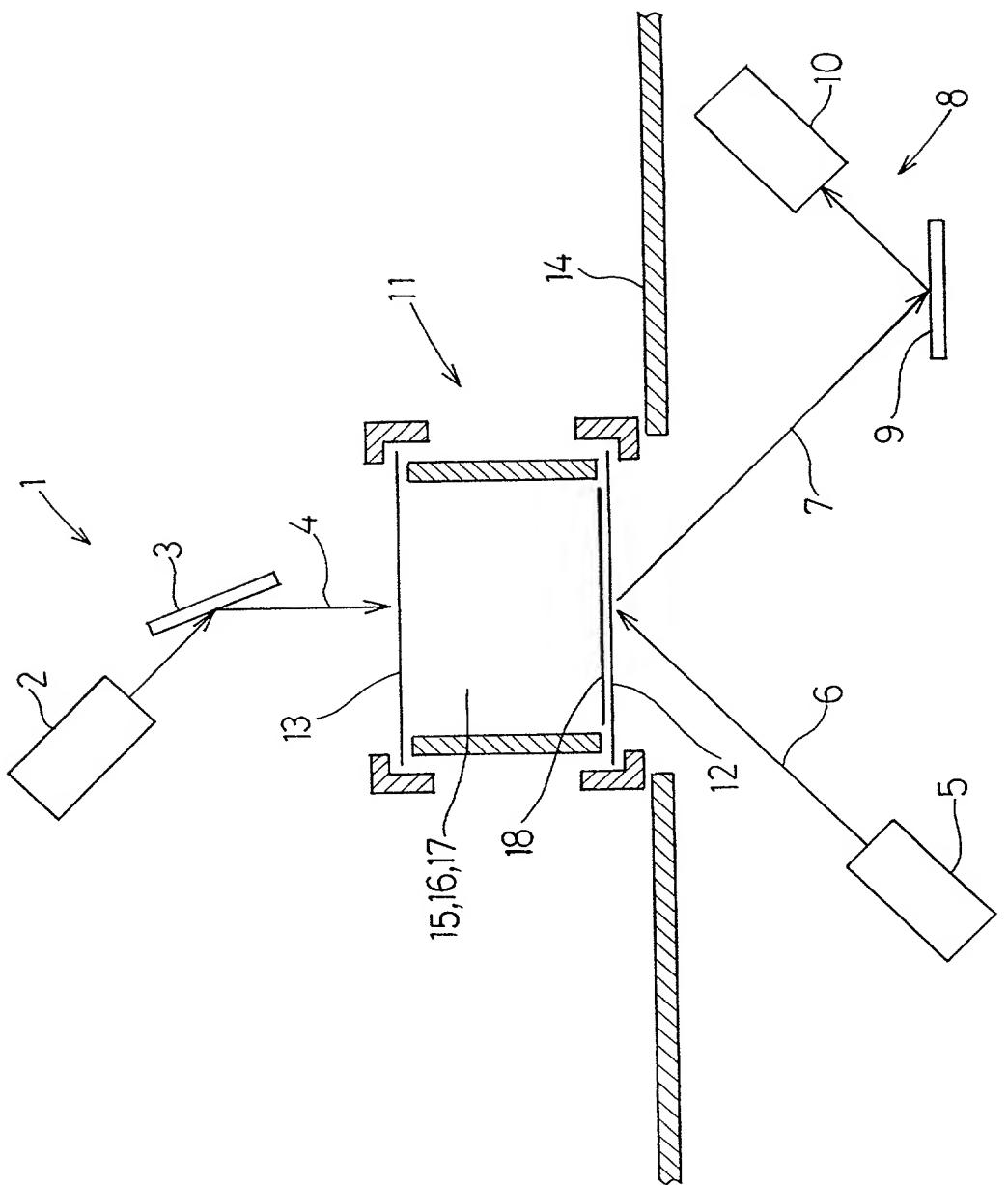


Fig. 4

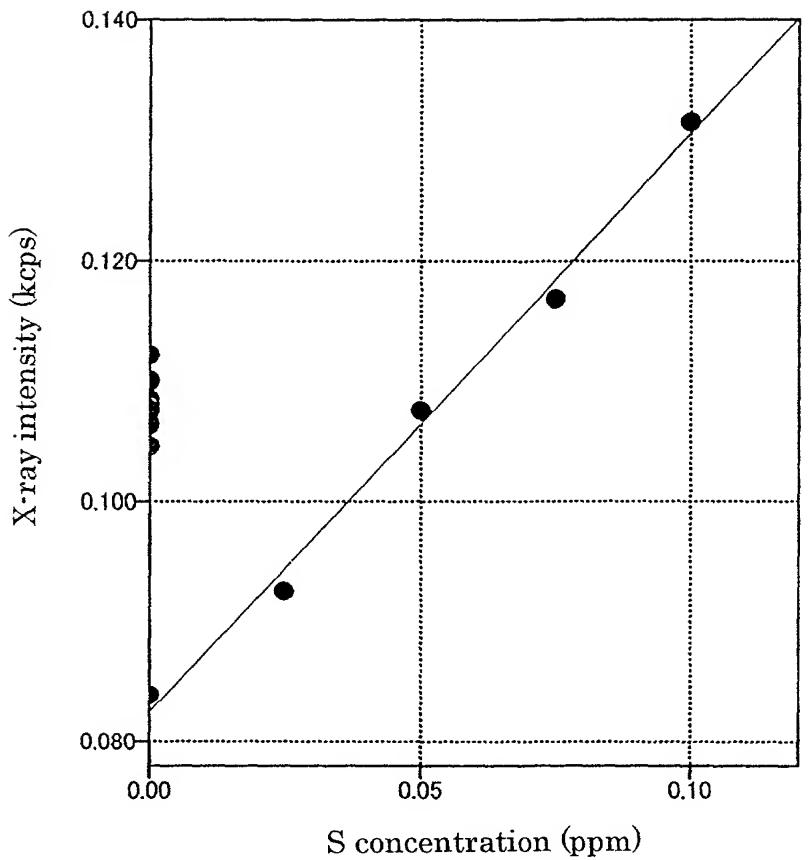


Fig.5

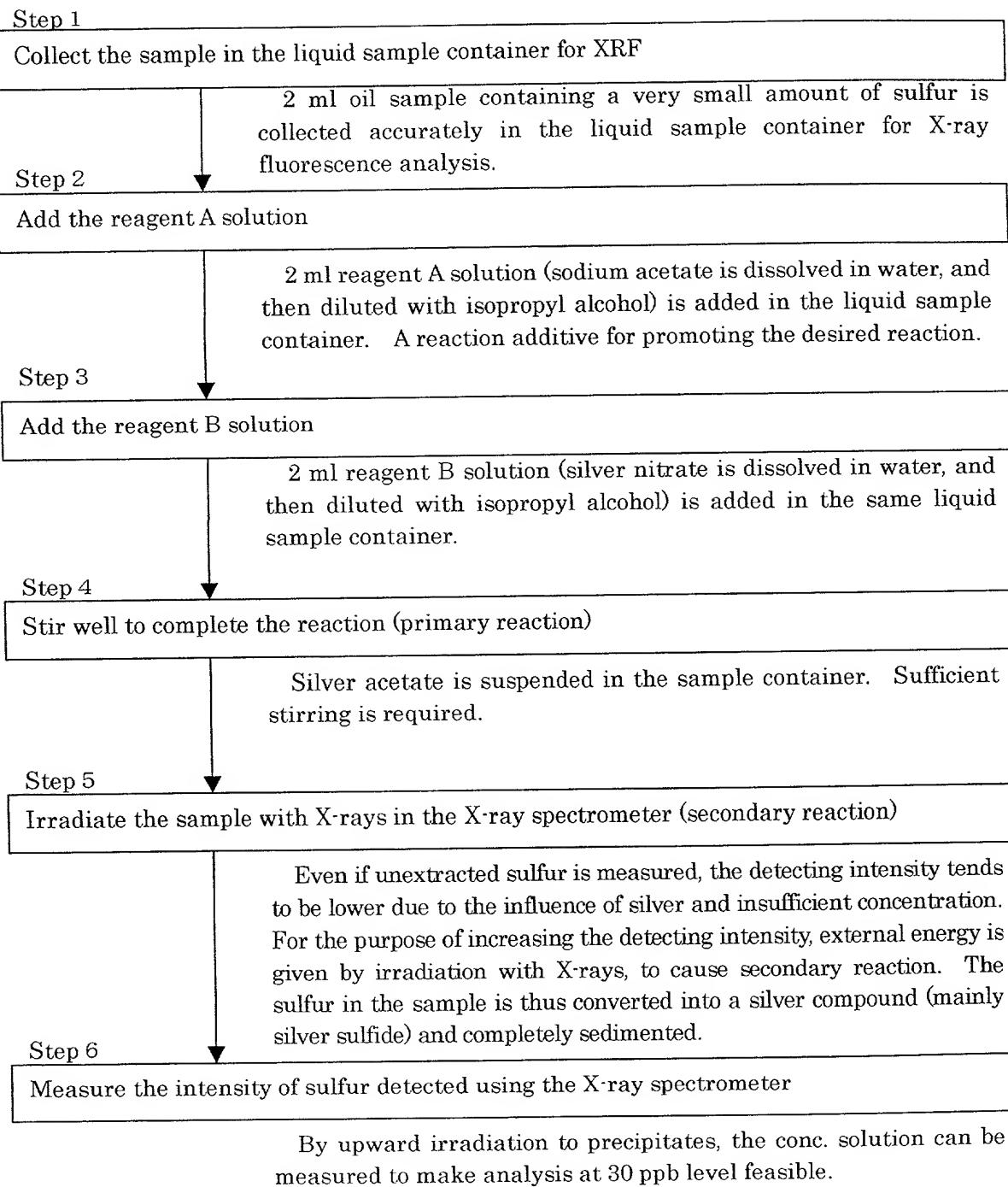


Fig. 6

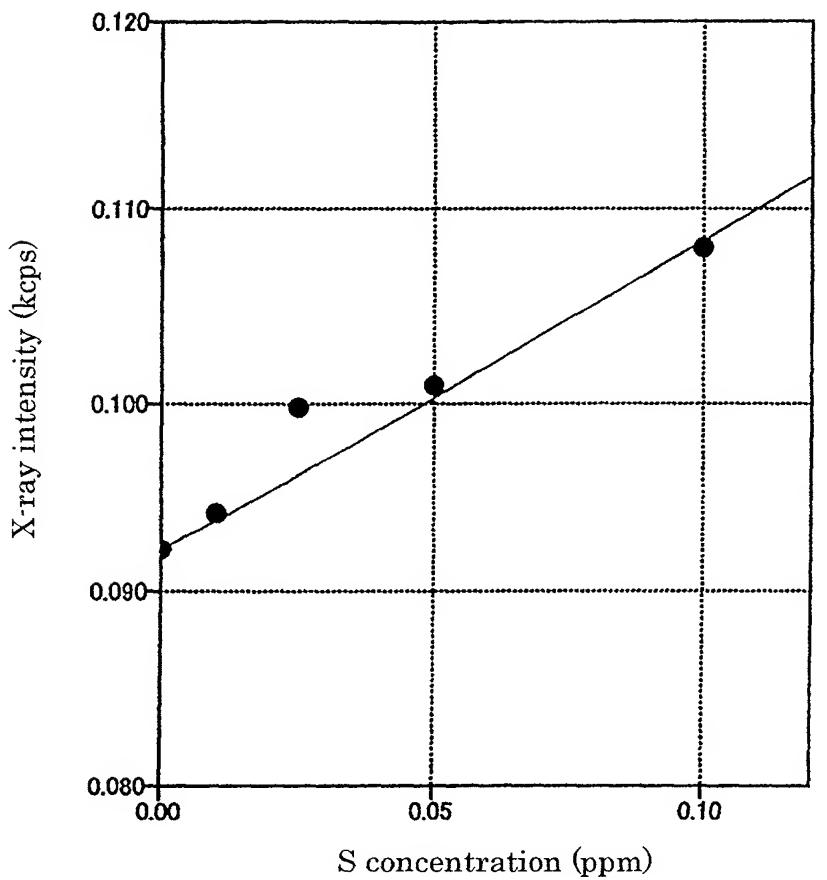


Fig. 7

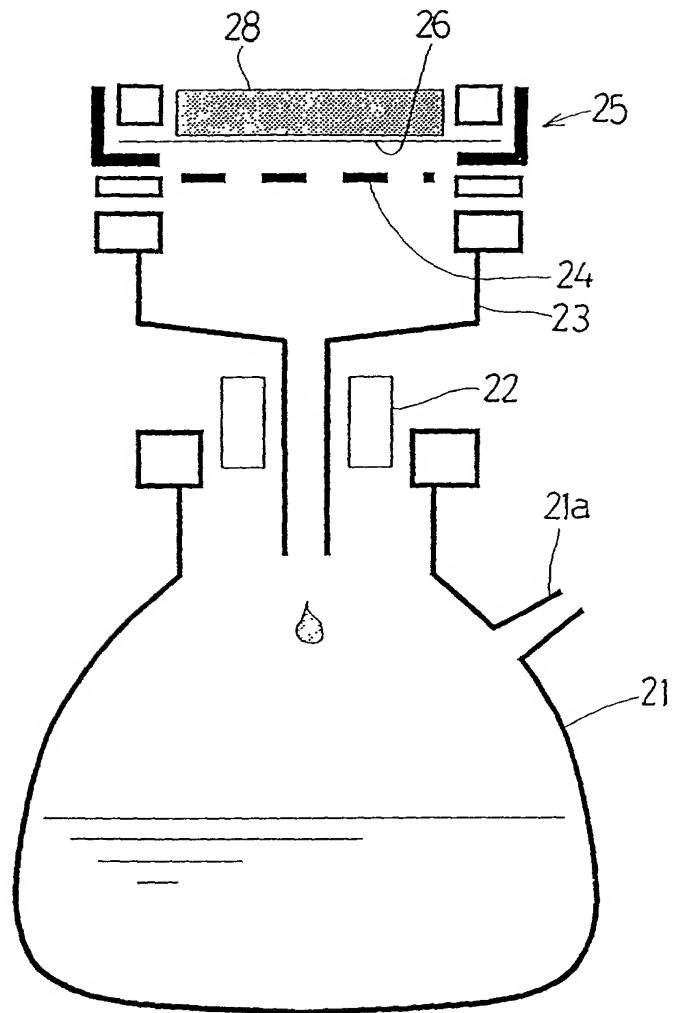


Fig. 8

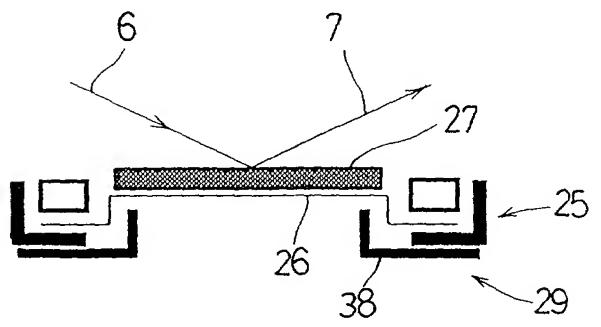


Fig. 9

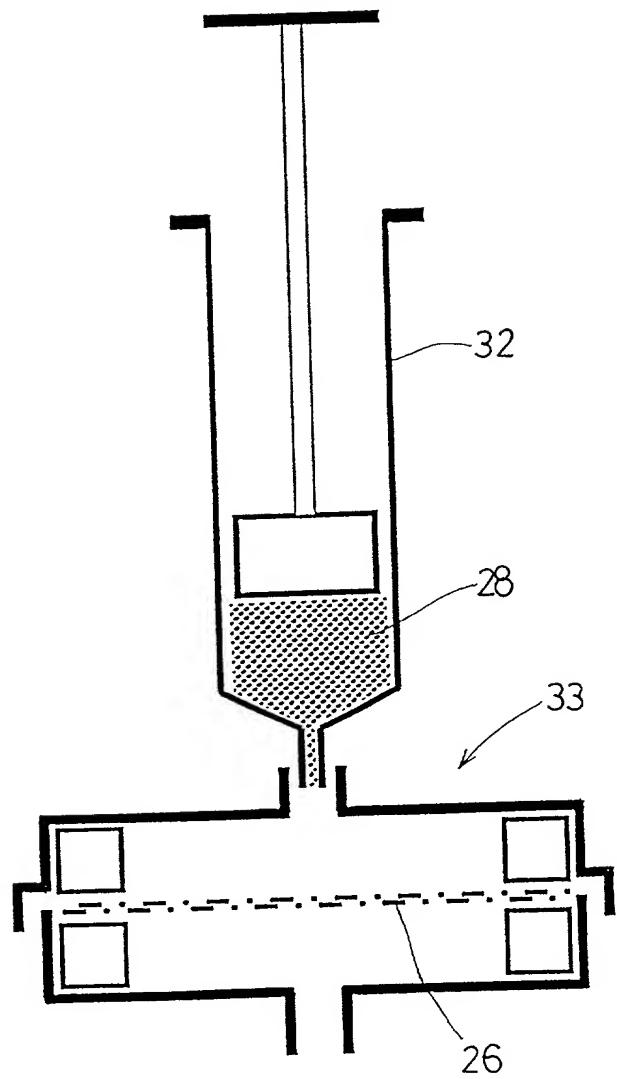


Fig. 10

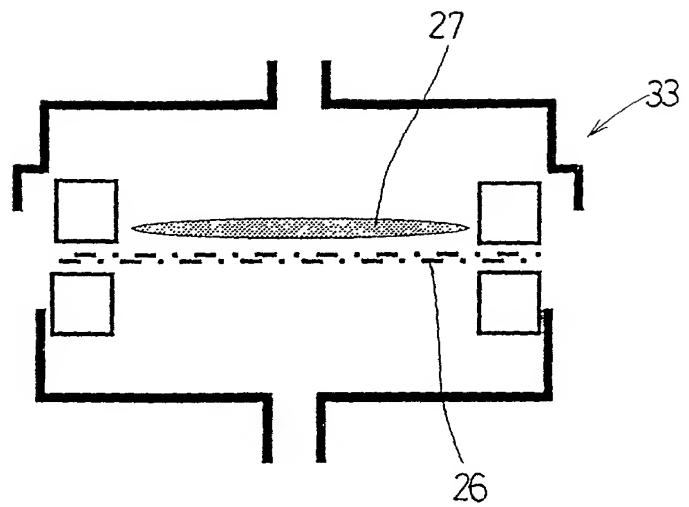


Fig. 11

